

## PATENT SPECIFICATION

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## COMPLETE SPECIFICATION

## Process for preparing Polyolefins

We, **FARBWERKE HOECHST ATKINGESSELL-SHAFT**, formerly Meister Lucius & Brüning, a Company recognised by German Law, of 6230 Frankfurt (Main) 80, Postfach 80 03 20, Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention provides polyolefins having a high stress cracking resistance, and a process for preparing them.

The production of bottles, cable sheathings and pipes by the process of extrusion of polyethylene or copolymers of ethylene with one or more other monomers requires polymers that have a broad molecular weight distribution, since polyolefins having a narrow molecular weight distribution show irregular surfaces on extruded articles (melt fracture) at a high throughput of the extruder. The material to be extruded into bottles, pipes and cable sheathings, must, furthermore, possess a sufficient stress cracking resistance.

It is known that the stress cracking resistance of a polyethylene can be improved by increasing its mean molecular weight and/or reducing its density. However too great an increase in the mean molecular weight is unsuitable, since the flow properties of a polymer are reduced when the molecular weight is raised, and therefore the throughput of the extruding machines is also reduced. When the density is decreased, by using instead of polyethylene itself a copolymer of ethylene with another  $\alpha$ -olefin the stress cracking resistance is improved but the stiffness, the ultimate tensile strength, the surface hardness and the softening point are reduced.

It was therefore necessary to find a process for preparing a polyethylene or a copolymer of ethylene with one or more other monomers which has a high density and a high stress

cracking resistance and shows an unobjectionable surface on an article when extruded at a high throughput. It has been proposed to prepare polymers having particular properties by carrying out the polymerization in several steps under different polymerization conditions.

British Patent No. 978,893 discloses a process for preparing polymers having a high impact strength and a high melting temperature by polymerizing olefins having from 2 to 18 carbon atoms, according to the Ziegler low-pressure-polymerization process, in two steps. In the first step, a polyolefin having a rigid nature is prepared and in the second step, a polymer having a predominantly soft nature is obtained. The second process step is preferably carried out without controlling the molecular weight. As catalysts suitable for the process, there may be mentioned salts of elements of groups 4 to 6 of the Periodic Table according to Mendelēef, and, as activators, organometallic compounds of the group Ia, IIa or IIIb of the Periodic Table according to Mendelēef.

According to British Patent No. 1,057,728, a continuous polymerization of ethylene, propylene or butene-(1) is carried out in the presence of a catalyst system consisting of an organic compound of a metal of groups 1 to 3 of the Periodic Table and a compound of a transition metal of groups 4 to 6 or 8 of the Periodic Table in an apparatus comprising at least two reaction zones.

In the separate zones, polymers having different molecular weights are prepared by carrying out the polymerization or copolymerization under different reaction conditions. The differing molecular weights are obtained by using different amounts of hydrogen and/or different catalyst concentrations and/or different temperatures, at least two reaction conditions having to be changed.

The polymer mixtures prepared by this

polymerization in several zones are distinguished by an improved processability. The same effect cannot be produced by subsequently mixing polymers having widely differing viscosities. The process, however, has the great drawback that several reaction conditions have to be changed during the polymerization.

The present invention provides a process for the preparation of a homo- or co-polymer of ethylene by a gaseous phase polymerization, or by a suspension polymerization in which the dispersion medium is in contact with a gaseous phase, of ethylene or a mixture comprising ethylene and up to 10% by weight of an  $\alpha$ -olefin that contains from 3 to 15 carbon atoms, which process comprises preparing from 5% to 30% by weight of the total polymer in the presence of from 0% to 10% of hydrogen, calculated on the total volume of the gaseous phase, and preparing from 70% to 95% by weight of the total polymer in the presence of from 20% to 80% of hydrogen, calculated on the total volume of the gaseous phase, both stages of the polymerization being carried out at a temperature within the range of 50°C to 120°C, preferably from 70°C to 90°C, and a pressure of up to 10 atmospheres gauge, preferably within the range of from 2 to 6 atmospheres gauge, and in the presence of a catalyst which is present in the first stage in an amount sufficient for both stages, said catalyst comprising

- a) in the case of a suspension polymerization, from 0.05 to 0.3 millimol per litre of dispersion medium, or in the case of a polymerization in the gaseous phase, from 0.05 to 0.3 millimol per 0.5 litre of reactor volume, of a trivalent titanium compound that contains chlorine, and
- b) from 0.1 to 3.0 millimols of aluminium per litre of dispersion medium or reactor volume, in the form of an aluminium trialkyl having the general formula  $AlR_3$ , in which each R represents a hydrocarbon radical that contains from 4 to 40 carbon atoms, or in the form of the reaction product of an aluminium trialkyl or an aluminium alkyl hydride with a dielectric that contains from 4 to 20 carbon atoms.

In the process of the present invention, the following embodiments are preferred:

- (a) from 5% to 30%, by weight of the total polymer is prepared by copolymerizing a mixture of ethylene and up to 10% by weight of the higher  $\alpha$ -olefin at a hydrogen content on the gaseous phase within the range of from 0% to 10% by volume, and from 70% to 95% by weight of the total polymer is prepared by polymerizing ethylene alone at a hydrogen content in the gaseous phase within the range of from 20% to 80% by volume.
- (b) From 5% to 30%, by weight of the total polymer is prepared by copolymerizing

a mixture of ethylene and up to 10% by weight of the higher  $\alpha$ -olefin at a hydrogen content on the gaseous phase within the range of from 0% to 10% by volume, and from 70% to 95% by weight of the total polymer is prepared by copolymerizing a mixture of ethylene and less than 10% by weight of the higher  $\alpha$ -olefin at a hydrogen content in the gaseous phase within the range of from 20% to 80% by volume, in which latter mixture the proportion of the higher  $\alpha$ -olefin is less than that in the first mixture specified above.

The polymerization may be carried out as a suspension polymerization in an inert dispersion medium, for example, an aliphatic or cycloaliphatic hydrocarbon or diesel oil, or may be carried out in the gaseous phase. In the former case the inert dispersion medium should not completely fill the reactor.

The titanium component of the catalyst, for example titanium trichloride or titanium alkoxy-chloride, is a solid and may be prepared by reducing titanium tetrachloride or, preferably, a chloro-alkoxy-titanate of the formula  $Ti(OR')_n-Cl$ , in which n is an integer from 1 to 4 and R' represents a hydrocarbon radical that contains from 2 to 3 carbon atoms, by means of alkyl-aluminium sesquichloride, dialkyl-aluminium monochloride, aluminium isoprenyl or a mixture thereof in an inert dispersing agent at a temperature within the range of from -60°C to +70°C, preferably from -30°C to 0°C, and if desired subjecting the product to a thermal treatment at a temperature within the range of from 60°C to 150°C and subsequently washing it with an inert dispersing agent.

When the polymerization is carried out in the gaseous phase, the volume of the gaseous phase is equal to volume of the reactor less the combined volume of the titanium component of the catalyst (which is a solid) and the polymer.

In the first of the process steps, the polymerization is effected at a hydrogen content in the gaseous phase ranging from 0% to 10% by volume, and yields, in general, polymers having a reduced viscosity, expressed in dl/gm, within the range of from 30 to 5 (measured in a 0.1% decahydronaphthalene solution at 135°C). The amount of the polymer proportion prepared in this step is in the range of from 5 to 30% by weight, calculated on the final polymer. In the other step, the polymerization is effected at a hydrogen content in the gaseous phase ranging from 20% to 80% by volume. It has been established that, under these conditions, polyethylenes or copolymers of ethylene and up to 10% by weight of the higher  $\alpha$ -olefins are obtained that have a reduced viscosity, expressed in dl/gm and measured as above, in the range of from 4.6 to 1.5.

Consequently according to the process of

1,174,542

3

- the invention, a polymer having a reduced viscosity, expressed in dl/gm and measured as above, in the range of from 1.5 to 4.6, is prepared in the presence of a polymer having a reduced viscosity, expressed in dl/gm and measured as above, in the range of from 5 to 30, to yield a homogeneous polymer. The final polymer therefore has an extremely broad molecular weight distribution.
- Compared with shaped articles made from copolymers of ethylene and  $\alpha$ -olefins, in which the hydrogen content in the gaseous phase has not been changed during the polymerization, shaped articles made from polymers of the invention, having the same density and the same reduced viscosity, show improved surfaces even with an increased throughput of the extruder.
- The process of the invention, moreover, provides polymers in which the higher molecular weight polymer chains also have a higher degree of branching.
- Owing to their special structure (extremely broad molecular weight distribution and higher degree of branching of the high molecular weight proportion), the polymers of the invention are especially suitable for producing bottles, pipes, cables and sheets or films having a high stress cracking resistance.
- The stress cracking resistance or the susceptibility to stress cracking may be measured according to ASTM D 1693—59T (Bell-test). Having a density of 0.950 gram.centimetre<sup>-3</sup> and a reduced viscosity, measured as above, of 2.7 dl/gm, shaped articles made from copolymers of ethylene and  $\alpha$ -olefins having a statistical incorporation of the comonomer (prepared by a uniform  $\alpha$ -olefin feed during the whole polymerization), show values of 180 hours in the Bell-test.
- In contrast thereto, shaped articles made from polymers in which the  $\alpha$ -olefin is predominantly incorporated into the higher molecular weight proportion, show values of more than 500 hours in the Bell-test, although their density and viscosity are the same as those of the above-mentioned products; Bell-test values of 180 hours have been achieved by shaped articles having a density of 0.956 gram.centimetre<sup>-3</sup>. Having the same stress cracking resistance as the above-mentioned articles, the shaped articles produced according to the invention show a higher density. In comparison with the copolymers of ethylene and  $\alpha$ -olefins having a statistical incorporation of the comonomer, the advantages of the polymers of the invention are an improved stiffness, a higher ultimate tensile strength, a better surface hardness and a higher softening point.
- The advantages of the process of the invention compared with those of hitherto proposed two-step polymerization processes (c.f. British Patent Specifications Nos. 978,893 and 1,057,728) consist in carrying out the present process in an especially simple technical manner. When aluminium trialkyls of the formula  $AlR_3$ , in which R represents a hydrocarbon radical containing from 4 to 40 carbon atoms, or, preferably reaction products of aluminium trialkyls or aluminium alkyl-hydrides with diolefins containing from 4 to 20 carbon atoms, such as aluminium isoprenyl, are used, high polymer yields exceeding 1 kilogram of polymer per 1 millimol of titanium compound are obtained with the catalyst concentration and under the reaction conditions mentioned above. The ash content is so low (0.03% by weight) that the polymers can be processed without first eliminating the catalyst.
- Therefore, the process of the invention provides, in a technically simple manner, polymers in which four structural parameters (molecular weight, molecular weight distribution, density and distribution of the comonomer) can be varied as desired so that the properties desired for the processing or the application of the products can be controlled.
- A greater control of the molecular weight distribution is obtained if the proportion of hydrogen in the gaseous phase is maintained as constant as possible throughout the course of each individual step.
- The process of the invention can be carried out continuously or discontinuously. The continuous method is carried out in two reactors arranged in series, by operating in the various polymerization vessels according to the conditions specified for the two steps.
- The following Examples illustrate the present invention:
- In each Example, when the polymerization had ceased, the polymer suspension was filtered under nitrogen over a pressure filter and the polymer was dried by passing over it nitrogen heated to 120°C.
- EXAMPLE 1**
- (a) Preparation of the titanium catalyst:
- A four-necked flask having a capacity of 500 millilitres was filled, under exclusion of air and moisture, with 100 millilitres of an olefin-free diesel oil fraction boiling in the range of from 130°C to 170°C, and 59.4 grams (240 millimols) of ethylaluminium sesquichloride. At 0°C and within 120 minutes, 37.6 grams (160 millimols) of dichloro-titanic acid diisopropyl ester in 120 millilitres of cyclohexane were added thereto dropwise while stirring. A fine brown precipitate separated. To allow for an after-reaction, the batch was maintained for 4 hours at 0°C while stirring. Subsequently, the mother liquor was decanted and the titanium residue was washed four times, each time with 200 millilitres of the above-mentioned diesel oil fraction.
- (b) Preparation of a copolymer of ethylene and butene-1 having a density of 0.957 gram.centimetre<sup>-3</sup> (two-step polymerization):
- 100 litres of diesel oil boiling in the range of from 130°C to 170°C, were introduced into

1,174,542

4

a vessel having a capacity of 150 litres, and the air was removed by flushing with pure nitrogen. The contents of the vessel were heated to 85°C and saturated with ethylene.

- 5 After 50 millilitres of aluminium isoprenyl (prepared according to Belgian Patent No. 601,855) and 20 millimols of a titanium catalyst (prepared as disclosed under 1 a)) had been added thereto, there were introduced, at 10 85°C within 90 minutes, 8 kilograms of ethylene, 0.13 kilogram of butene-(1), and such an amount of hydrogen that the hydrogen content in the gaseous phase was 2% by volume. A polymer sample showed a reduced viscosity of 9.3 dl/gm (measured in a 0.1% decahydronaphthalene solution at 135°C).

- 15 At the same temperature and within 6 hours, there were introduced 32 kilograms of ethylene and such an amount of hydrogen that the hydrogen content in the gaseous phase was 45% by volume. During the polymerization, the maximum pressure in the vessel was 6 atmospheres gauge.

- 20 There were obtained 40 kilograms of a copolymer having a reduced viscosity of 2.9 dl/gm (measured in a 0.1% decahydronaphthalene solution at 135°C) and a density of 0.957 gram-centimetre<sup>-3</sup>.

#### COMPARISON EXAMPLE 1 (single-step polymerization)

- 30 In a comparative test, a copolymer having a density of 0.957 gram-centimetre<sup>-3</sup>, was prepared in a single-step polymerization by continuously adding butene-(1).

- 35 To a vessel having a capacity of 150 litres there were added 100 litres of diesel oil having a boiling range of from 130°C to 170°C, and the vessel was flushed with nitrogen. The contents of the vessel were heated to 85°C and saturated with ethylene.

- 40 After 50 millilitres of aluminium isoprenyl and 20 millimols of a titanium catalyst (prepared as disclosed under a) in Example 1), had been added thereto, there were introduced into the vessel at 85°C ethylene, 0.2% by weight of butene-(1) and such an amount of hydrogen that the hydrogen content in the gaseous phase was 35% by volume.

- 45 Under these conditions, the polymerization was carried out for 8 hours under a pressure of 6 atmospheres gauge. There were obtained 44 kilograms of a copolymer having a reduced viscosity of 2.9 dl/gm (measured in a 0.1% decahydronaphthalene solution at 135°C), and 50 a density of 0.957 gram-centimetre<sup>-3</sup>.

#### COMPARISON EXAMPLE 2 (single-step polymerization)

- 50 Under the conditions disclosed in comparative Example 1, 40 kilograms of ethylene were polymerized with 0.13 kilogram of butene-(1) (=0.33% by weight) to yield a copolymer having a reduced viscosity of 2.9

dl/gm (measured as above, and a density of 0.953 gram-centimetre<sup>-3</sup>.

The properties of the copolymers are compared with each other in Table 1.

#### EXAMPLE 2

Preparation of a copolymer of ethylene and butene-(1) having a density of 0.948 gram-centimetre<sup>-3</sup> (two-step polymerization).

100 Litres of diesel oil having a boiling range of from 130°C to 170°C were introduced into a vessel having a capacity of 150 litres; air was removed by flushing with pure nitrogen. The contents of the vessel were heated to 85°C and saturated with ethylene. After 50 millilitres of aluminium isoprenyl and 20 millimols of a titanium catalyst (prepared as disclosed under 1 a)) had been added thereto, there were introduced into the vessel, at 85°C and within 60 minutes, 6 kilograms of ethylene, 0.52 kilogram of butene-(1); and such an amount of hydrogen that the hydrogen content in the gaseous phase was 0.2% by volume. A polymer sample showed a reduced specific viscosity of 23.6.

At the same temperature and within 6 hours, there were then introduced into the vessel 36 kilograms of ethylene and such an amount of hydrogen that the hydrogen content in the gaseous phase was 50% by volume. During the polymerization, the maximum pressure in the vessel was 6 atmospheres gauge. There were obtained 42 kilograms of a copolymer having a reduced viscosity of 2.8 dl/gm (measured as in Example 1); and a density of 0.948 gram-centimetre<sup>-3</sup>.

#### COMPARISON EXAMPLE 3 (single-step polymerization)

In a comparative test, a copolymer having a density of 0.948 gram-centimetre<sup>-3</sup>, was prepared in a single-step polymerization process by continuously adding butene-(1).

To a vessel having a capacity of 150 litres there were added 100 litres of diesel oil having a boiling range of from 130°C to 170°C, and the vessel was flushed with nitrogen. The contents of the vessel were heated to 85°C and saturated with ethylene.

After 50 millilitres of aluminium isoprenyl and 20 millimols of a titanium catalyst (prepared as disclosed under 1 a)) had been added thereto, there were introduced at 85°C, ethylene, butene-(1) (0.9% by weight, calculated on the ethylene used); and such an amount of hydrogen that the hydrogen content in the gaseous phase was 36% by volume. The polymerization was carried out within 8 hours under a pressure of 6 atmospheres gauge. There were obtained 44 kilograms of a copolymer having a reduced viscosity of 2.8 dl/gm (measured as in Example 1) and a density of 0.948 gram-centimetre<sup>-3</sup>.

#### EXAMPLE 3

An autoclave having a capacity of 1 litre

1,174,542

5

5 was charged with 0.5 litre hexane, 20 millimols  
aluminium tri-isobutyl and 2.5 millimols of  
the titanium catalyst used in Example 1. Air  
was removed by flushing with nitrogen, hydro-  
10 gen was added to a pressure of 4 atmospheres,  
and the autoclave was heated to 85°C. Over a  
period of 30 minutes a total of 300 gms.  
ethylene were added at a total pressure of 7  
atmospheres. After releasing the pressure and  
15 evaporating the solvent there were obtained  
300 gms. polymer that contained the whole  
of the catalyst.

The polymer and catalyst was added to a  
horizontal reactor, hydrogen was added to a  
15 pressure of 4 atmospheres, and the tempera-  
ture was raised to 85°C. 3.45 Kgm. ethylene  
were added, with stirring, over a period of 7  
hours at this temperature, and the pressure  
was then released.

The reactor contained 3.75 kilograms of 20  
polyethylene which had a reduced viscosity of  
1.8 dl/gm (measured in a 0.1% decahydro-  
naphthalene solution at 135°C).

To the polymer in the reactor there were 25  
added a further 1.2 kilograms ethylene and  
50 grams 1-butene, the addition being carried  
out over a period of 1.5 hours at a tempera-  
ture of 85°C. The pressure was again released  
and there were obtained 5 kilograms of a co-  
polymer having a reduced viscosity of 3.0 30  
dl/gm (measured in a 0.1% decahydroph-  
thalene solution at 135°C) and a density of  
0.949 gram.centrimeter<sup>-3</sup>.

The properties of the copolymers prepared 35  
in the foregoing Examples and Comparison  
Examples are compared in Table 1.

TABLE I

Copolymer prepared according to	Density gram./centimetre <sup>3</sup> (buoyancy method)	Reduced viscosity in 0.1%-decylhydronaphthalene solution at 135° C. dl./gm.	Ball indentation hardness 10 seconds VDE 0302	
Example 1 comparison Example 1 comparison Example 2 Example 2 comparison Example 3 Example 3	0.957	2.9	580	
	0.957	2.9	580	
	0.953	2.9	530	
	0.948	2.8	480	
	0.948	2.8	480	
	0.949	3.0		
	Crystalline melt point (polarization microscope) °C	Stress cracking resistance Bell test (hours) ASTMD 1693-59T	Surfaces of bottles (produced under the same conditions)	Relative throughput in extrusion pro- cessing under the same conditions
Example 1 comparison Example 1 comparison Example 2	130 — 132	220	smooth	1
	130 — 132	25	rough; light melt fracture	0.8
	128 — 131	60	rough; light melt fracture	0.83
Example 2 comparison Example 3	126 — 129	~500	smooth	1.3
	126 — 129	120	rough; melt fracture	0.82

1,174,542

7

## WHAT WE CLAIM IS:—

1. A process for the preparation of a homo- or co-polymer of ethylene by a gaseous phase polymerization, or by a suspension polymerization in which the dispersion medium is in contact with a gaseous phase, of ethylene or a mixture comprising ethylene and up to 10% by weight of an  $\alpha$ -olefin that contains from 3 to 15 carbon atoms, which process comprises preparing from 5% to 30% by weight of the total polymer in the presence of from 0% to 10% of hydrogen, calculated on the total volume of the gaseous phase, and preparing from 70% to 95% by weight of the total polymer in the presence of from 20% to 80% of hydrogen, calculated on the total volume of the gaseous phase, both stages of the polymerization being carried out at a temperature within the range of from 50°C to 120°C and a pressure of up to 10 atmospheres gauge, in the presence of a catalyst which is present in the first stage in an amount sufficient for both stages, said catalyst comprising
- a) in the case of a suspension polymerization, from 0.05 to 0.3 millimol per litre of dispersion medium, or in the case of a polymerization in the gaseous phase, from 0.05 to 0.3 millimol per 0.5 litre of reactor volume, of a trivalent titanium compound that contains chlorine, and
  - b) from 0.1 to 3.0 millimols of aluminium per litre of dispersion medium or reactor volume, in the form of an aluminium trialkyl having the general formula  $AlR_3$ , in which each R represents a hydrocarbon radical that contains from 4 to 40 carbon atoms, or in the form of the reaction product of an aluminium trialkyl or an aluminium alkyl hydride with a diolefin that contains from 4 to 20 carbon atoms.
2. A process as claimed in claim 1, wherein from 5% to 30% by weight of the total polymer is prepared by copolymerizing a mixture

of ethylene and up to 10% by weight of the higher  $\alpha$ -olefin in the presence of from 0% to 10% of hydrogen, calculated on the total volume of the gaseous phase, and from 70% to 95% by weight of the total polymer is prepared by polymerizing ethylene alone in the presence of from 20% to 80% of hydrogen, calculated on the total volume of the gaseous phase.

3. A process as claimed in claim 1, wherein from 5% to 30% by weight of the total polymer is prepared by copolymerizing a mixture of ethylene and up to 10% by weight of the higher  $\alpha$ -olefin in the presence of from 0% to 10% of hydrogen, calculated on the total volume of the gaseous phase, and from 70% to 95% by weight of the total polymer is prepared by copolymerizing a mixture ethylene and the higher  $\alpha$ -olefin in the presence of from 20% to 80% of hydrogen, calculated on the total volume of the gaseous phase, in which latter mixture the proportion of the  $\alpha$ -olefin is less than that in the first mixture specified above.

4. A process as claimed in any one of claims 1 to 3, wherein the polymerization is carried out at a temperature within the range of from 70°C to 90°C.

5. A process as claimed in any one of claims 1 to 4, wherein the polymerization is carried out under a pressure within the range of from 2 to 6 atmospheres gauge.

6. A process as claimed in any one of claims 1 to 5, wherein as component a) of catalyst there is used titanium trichloride or titanium alkoxychloride.

7. A homo- or co-polymer of ethylene whenever obtained by a process as claimed in any one of claims 1 to 6.

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